SILICON NITRIDE WHISKER REINFORCED GLASS MATRIX COMPOSITES

RIGHTS OF THE GOVERNMENT

BACKGROUND OF THE INVENTION

This invention relates to a fiber reinforced structural material. More specifically, this invention relates to a composite structure comprised of high strength silicon nitride whiskers in a glass matrix.

Fiber reinforced organic matrix composites are widely used and accepted as structural materials because of their desirable attributes of high strength high modulii and low density. In general, most of these composites comprise an organic polymer matrix, such as an epoxy resin, a polyimide, a polycarbonate, or similar material. The matrices are reinforced with a wide variety of fibers including glass, carbon, graphite and boron. However, even the best of these composites are limited to an operational temperature below about 600° F. (300° C.).

The severe environment encountered by advanced missile systems precludes the use of organic matrices. Radomes for such systems must have acceptable resistance to rain and particle erosion as well as high thermal stability and thermal shock resistance. Generally, ceramic materials meet one or more of these requirements. One further requirement for radomes, that being transparency to X band radiation, precludes the use of certain ceramic materials. Silicon carbide yarn reinforced glass and glass ceramic composites, although very strong, tough, and environmentally stable, have been found to be essentially opaque to X band radiation. Other materials, such as boron nitride reinforced glass and glass ceramic composites, are X band transparent, but are extremely weak and brittle.

Thus, what is desired is a composite material which exhibits superior strength and toughness, high thermal stability and is transparent to X band radiation.

Accordingly, it is an object of the present invention to provide an improved silicon nitride whisker, glass matrix composite material.

Another object of the present invention is to provide a process for making an improved silicon nitride whisker glass matrix composite material.

Other objects and advantages of the present invention will become apprent to those skilled in the art from a reading of the following detailed disclosure.

DESCRIPTION OF THE INVENTION

In accordance with the present invention there is provided a composite material consisting of silicon nitride whiskers dispersed through a glass matrix.

This improved composite material is prepared by the steps of:

(a) dispersing silicon nitride whiskers in a suitable liquid and separating off the fines;

(b) removing the liquid from the remaining silicon nitride whiskers;

(c) blending a desired quantity of the whiskers from step (b) with a desired quantity of glass frit and a temporary binder in a suitable liquid to form a mixture;

(d) coating a suitable carrier substrate with a layer of the mixture;

(e) evaporating the liquid out of the layer;

(f) separating the thus-dried layer from the carrier; and

(g) stacking a plurality of such dried layers in a jig and hot-pressing the layers together at a temperature and pressure sufficient to cause the glass particles to flow to form a void-free matrix throughout which are dispersed the silicon nitride whiskers. The temporary binder is burnt out during the hot-pressing operation, or in a separate heat treatment prior to hot pressing.

Silicon nitride whiskers are available commercially from Versar Incorporated, Springfield, VA and Tateho Chemical Industries, Hyoga-Ken, Japan. The Versar whiskers are in the form of intergrown mats consisting of whiskers having diameters ranging from under 1 micron to about 20 microns, with lengths ranging from several microns to several inches. The Tateho whiskers generally have diameters ranging from 0.2 to 0.5 microns and lengths ranging from about 50 to 300 microns.

The silicon nitride whiskers are processed by first dispersing a quantity of whiskers in a suitable liquid, such as water at a relatively low shear, to avoid damaging the whiskers. In general, about 2 g. of whiskers per liter of water will provide sufficient volume to ensure separation of the whiskers. A blender may be used to disperse the whiskers. Generally about 1 to 5 minutes at low speed is sufficient to disperse the whiskers.

The whiskers may then be treated with hydrofluoric acid to remove silica or silicate impurities. It is convenient to combine several batches of the whiskers in water dispersions in a large container and add about 50 to 100 ml of concentrated HF per liter of the whisker/-water dispersion with mixing. This mixture, after stirring is allowed to settle about 30 to 90 minutes. The liquid above the settled-out whiskers is then removed. The remaining whisker mass is mixed with distilled water, allowed to settle, as above, and the liquid removed. This washing step is repeated 1 to 4 times to ensure removal of the HF. The remaining whisker mass is then filtered and dried.

A weighed portion of the dried whiskers is dispersed in water or a lower alkyl alcohol. A desired quantity of glass frit and a suitable temporary binder material is then added to the dispersion of whiskers. In general, the ratio of whiskers to glass, by weight can range from about 1:2 to 2:1, preferably about 1:2 to 1:1.2. The combined quantity of whiskers and glass should be about 3 to 8 weight percent of the resulting mixture, preferably about 5–6 weight percent. About 1 gram of the temporary binder material per gram of whiskers has been found to be satisfactory for later processing.

The mixture of whiskers, glass frit and temporary binder material is spread in a layer onto a carrier substrate and the liquid is evaporated to provide a precursor sheet.

The temporary binder material can be any material known in the art which is miscible with the liquid medium, i.e. water or a C1 to C3 alcohol, and which, when the liquid is removed, is solid at room temperature. A suitable binder for use with water medium is Rhoplex, an acrylic latex available from Rhom and Haas, Inc., Philadelphia, PA.

To make the silicon nitride whisker reinforced glass matrix of this invention, any of a large number of glasses may be used. Any of the glasses listed in Table I, for example, as well as others, may be used for the glass